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Corrosion of Roof Bolt Steels in Missouri Lead and Iron Mine Waters

By M. M. Tilman, A. F. Jolly III, and L. A. Neumeier

UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°F	degree Fahrenheit	nA/cm^2	nanoampere per square centimeter
h	hour	ppm	part per million
in	inch	V	volt
min	minute	wt pct	weight percent
mL	milliliter		
mpy	mil per year		

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CORROSION OF ROOF BOLT STEELS IN MISSOURI LEAD AND IRON MINE WATERS

By M. M. Tilman,[1] A. F. Jolly III,[2] and L. A. Neumeier[3]

ABSTRACT

As part of ongoing research to improve mine safety, the Bureau of Mines conducted research on the corrosion of friction rock stabilizer steels in five Missouri lead and iron mine waters. Electrochemical corrosion tests, including evaluation of pitting tendency, were performed on two types of high-strength, low-alloy (HSLA) steels and galvanized steel in four Missouri lead mine waters and one Missouri iron mine water. The tests were conducted at in-mine water temperatures in both air-saturated and deaerated waters. Static, weight-loss corrosion tests were also conducted on HSLA steel specimens in the five Missouri mine waters for 2,900-h duration at average in-mine water temperatures and air-dissolved oxygen contents of 6 to 7 ppm. Corrosion rates determined by the weight-loss tests were roughly comparable with rates determined electrochemically in deaerated waters containing 0.3 to 0.5 ppm dissolved oxygen content. Passivation of specimen (nongalvanized) surfaces in air-saturated waters resulted in very low electrochemically determined corrosion rates. Pitting tendency was generally higher for both HSLA steels in air-saturated waters than in deaerated waters. Galvanized steel generally exhibited higher tendency to pitting in the deaerated waters than in the aerated mine waters.

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INTRODUCTION

In the late 1970's, a different concept in roof-rock bolts was introduced commercially in friction rock stabilizers. Several types of the stabilizers have been developed and are in commercial use. They are thin-wall tubular devices which, unlike point-anchor bolts, exert their holding power by compressive forces acting over the length of the stabilizer. Because of their unique holding mechanism, the stabilizers are claimed to be particularly useful in softer rock such as sandstone and shale. However, because of the relatively thin wall, they are more vulnerable to corrosion, which can under certain conditions cause a serious loss of strength in a relatively short time. Friction rock stabilizers are normally not recommended for use where particularly long-term support is required.

The Split Set[4] stabilizer has gained wide acceptance in the metal mining industry. Over 25 million Split Set stabilizers have been installed, primarily in metal mines, not only in the United States but also in approximately 30 foreign countries. The Split Set stabilizer is a slotted tube 1-1/2 in or larger in diameter, which is forced into an undersize hole, with the result that compressive forces act over the entire length of the tube. The stabilizers are made of steel sheet nominally about 0.1 in thick and are produced in various lengths. Although a device has been developed to check for proper installation of Split Set stabilizers (9),[5] no technique has been developed for revealing loss of strength after installation, such as that due to corrosion. Two types of high-strength, low-alloy (HSLA) steels are used to manufacture Split Set stabilizers. Hot-dip-galvanized Split Set stabilizers are also available.

Research was previously conducted by the Bureau of Mines on the corrosion resistance of Split Set stabilizers in copper and uranium mine waters (11). Results of the prior research indicated that galvanized steel, not surprisingly, was far superior to the unprotected steel in general corrosion resistance. For the waters evaluated, limited corrosion tests indicated little tendency to pitting of galvanized steel compared to that for the uncoated steel. Copper-bearing HSLA steel was shown to be slightly more corrosion resistant than non-copper-bearing HSLA steel. Equations were developed relating dissolved oxygen, chloride, sulfate, and magnesium contents of the mine waters to corrosion rates of two nongalvanized HSLA steels. An equation was developed relating dissolved oxygen content and mine water temperature to corrosion rate of galvanized steel. The research indicated the need for prevention and control of corrosion of the roof support members in view of the considerable variables involved in prediction of corrosion damage.

The work with Missouri mine waters described in this report is an extension of the previous research on corrosion of Split Set stabilizers in Western mine waters and is part of the Bureau's ongoing program to improve mine safety. Results of the work are intended to aid Mine Safety and Health Administration and mining industry personnel in better predicting safe installation periods of friction rock stabilizers.

EXPERIMENTAL PROCEDURE

The Ingersoll-Rand Corp. provided sheet samples of EX-TEN-H60 and KAI-WELL-55, the two high-strength, low-alloy (HSLA) steels from which Split Set stabilizers are manufactured, for use in the corrosion experiments. Samples of the steels were analyzed for comparison with specifications. Microstructures of the steels were shown in a previous report (11). Galvanized plain-carbon steel sheet (flat), obtained from a commercial supplier, was used for corrosion testing

[4]Registered trademark of Ingersoll-Rand Co.

[5]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

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instead of galvanized Split Set stabilizer steel, because galvanized Split Set sheet stock was not readily available. Corrosion samples cut from galvanized stabilizers were incompatible with the corrosion test equipment because of their curvature; the specimen holder would only accept flat specimens for a proper fit. Attempts to flatten the galvanized stabilizer samples damaged the galvanized surface. The use of plain-carbon galvanized steel sheet was deemed acceptable, since variation in composition of the outer zinc layer of galvanized coatings, if present, has been indicated to be insignificant with respect to detrimental corrosion effects (2, p. 712; 3, p. 646). The scope of the research did not extend to the complex condition in which corrosion might, with extended time, proceed to penetrate the zinc coating. It should be mentioned that, even in that eventuality, the zinc continues to provide sacrificial protection to steel until considerable steel is exposed (2, p. 712).

A Swellex[6] bolt, another type of friction rock stabilizer, was provided by personnel at a western mine. A section of the bolt was flattened, corrosion samples punched, and the steel analyzed.

Five-gallon water samples were obtained from four Missouri lead mines and one Missouri iron mine. The lead mines were the Fletcher Mine, Indian Creek Mine, and St. Joe #28 Mine, of St. Joe Lead Co., and the Magmont Mine, of Cominco American/Dresser Industries. The iron mine was the Pea Ridge Mine of Pea Ridge Iron Ore Co. Mine water temperatures were measured at the sampling site. The samples were analyzed for common elements, and the pH values were measured. The Langelier (saturation) indices, which indicate the relative tendency for $CaCO_3$ to precipitate from water and form a protective deposit, were calculated using the values for the total dissolved solids, calcium and HCO_3 contents, and pH and temperature. Corrosion rates of EXTEN-H60 and KAI-WELL-55 steels were measured by both electrochemical and weight-loss methods in all five mine waters. Corrosion rates of galvanized steel were measured in the five mine waters by electrochemical methods only. Corrosion rates of the Swellex steel were measured electrochemically in air-saturated and deaerated water from the St. Joe #28 lead mine.

A commercial corrosion measurement system (10), consisting of a corrosion cell and microprocessor unit, was used to obtain "instantaneous" corrosion rates. In operation, the corrosion cell containing test solution, specimen, counter electrodes, and standard calomel reference electrode is immersed in a controlled-temperature water bath. The microprocessor unit, correctly programmed and with appropriate operator skill, calculates corrosion rates based on the corrosion current. Tafel constants obtained in anodic and cathodic polarization plots are used in a subsequent polarization resistance plot to calculate the general corrosion rate. A more detailed discussion of the electrochemical test equipment and technique may be found in a previous Bureau report (11). Discussions of electrochemical measurement of corrosion rates are found in several references (1, 4, 6, 8). Corrosion rates were determined electrochemically in both helium-degassed (deaerated) and air-saturated (aerated) mine waters at the measured in-mine water temperature.

In addition to corrosion rate determinations, pitting susceptibility scans were run on each steel in all except one mine water. The pitting scans were run in both deaerated and air-saturated waters at the in-mine water temperature.

Test specimens of the HSLA steels were ground over 600-grit SiC abrasive paper, ultrasonically cleaned in ethanol, and dried in a warm air blast. Galvanized specimens were prepared with the same procedure, except that grinding was omitted. After immersion in deaerated test solutions, the specimens were electrolytically cleaned by operating the cell with the specimen as cathode [-1.0 V vs standard calomel electrode (SCE)] for 1 min immediately preceding the test run. Specimens tested in aerated waters were not electrolytically cleaned because of the resulting instability of the open circuit potential (E_{corr}). A series of test runs

[6]Registered trademark of Atlas Copco.

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was made with EX-TEN-H60 steel in Magmont water to determine variation of the open circuit cell potential (corrosion potential) and corrosion rate, the latter as a function of the time interval in which the specimen was in solution prior to initiation of linear polarization tests.

Total immersion, weight-loss determination of corrosion rates from static tests was based on ASTM Standard G31-72, "Laboratory Immersion Corrosion Testing of Metals" (5). Specimens of EX-TEN-H60 and KAI-WELL-55 steels were machined from sheet stock to approximately 1 in square, and a 0.082-in-diam hole was drilled in each specimen for suspension in the test solution. The specimens were ground over 120-grit SiC paper, dimensions were measured, and surface areas calculated. They were then ultrasonically cleaned in ethanol and weighed. Two specimens of each steel were suspended with nylon line in each mine water. The four lead mine waters were contained in 1,000 mL beakers immersed in a constant-temperature bath at 62°±0.5° F. The temperature of 62° F was the average temperature of the lead mine waters, which ranged from 61° to 64° F. The beaker containing the iron mine water was immersed in a separate constant-temperature bath, which was maintained at 80°±0.5° F, the in-mine water temperature. Test solution temperatures were monitored daily. Dissolved oxygen contents were measured daily for the first few days and weekly thereafter for the duration of the test. No attempt was made to control dissolved oxygen content. The beakers were covered loosely to permit access of air. The total-immersion tests were terminated at 2,900 h. The specimens were rinsed and scrubbed with a bristle brush under running water to remove loosely adherent corrosion products, dried in a warm air blast, and weighed. Corrosion rates were calculated from weight losses.

RESULTS AND DISCUSSION

Results of chemical analyses of the HSLA steels used for the manufacture of Split Sets are shown in table 1. The EX-TEN-H60 composition was within specifications (12). No analysis was performed for the nonspecified elements of sulfur, phosphorus, and copper, nor for nitrogen. The composition of KAI-WELL-55 steel was within specifications (7) for those elements for which analyses were performed. No analyses were conducted on KAI-WELL-55 steel for the specified minor elements of sulfur and phosphorus. Results of analysis of the Swellex steel, in weight-percent, are as follows: C, 0.06; Mn, 0.24; Si, <0.05; S, 0.02; P, <0.01; and balance Fe.

TABLE 1. - High-strength, low-alloy steel compositions, weight percent

Element	EX-TEN-H60		KAI-WELL-55	
	Specification[1]	Analysis	Specification[2]	Analysis
C.....	Max 0.25	0.23	0.2 -0.3	0.3
Cb.....	([3])	.01	NS	NA
Cu.....	NS	NA	Min .20	.32
Mn.....	Max 1.35	1.22	.85-1.30	1.12
N.....	Max .012	NA	NS	NA
P.....	NS	NA	Max .05	NA
S.....	NS	NA	Max .05	NA
Si.....	NS	<.02	Max .12	<.02
V.....	([3])	.01	NS	NA

NA Not analyzed.
NS Not specified.
[1]Woldman (12), p. 435.
[2]Ingersoll-Rand Co. (7).
[3]Specification minimum 0.02 wt pct Cb + V.

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Analyses of mine waters are shown in table 2. All of the waters were somewhat basic, with pH values of 8.0 or 8.1. Calculated Langelier indices of -0.2 to +0.5 indicate little tendency toward carbonate precipitation for any of the five mine waters. The Pea Ridge water contains relatively large amounts of Na^+, SO_4^{-2}, and HCO_3^- ions, as reflected in the large amount of total dissolved solids. The Magmont and Indian Creek waters are much higher in Cl^- ion content than the other waters, and the Magmont water is also relatively high in HCO_3^- ion content. All of the waters analyzed low in iron contents, <0.1 ppm Fe.

Shown in figures 1 and 2 are examples of anodic and cathodic polarization plots, respectively, from which Tafel constants (slopes) are calculated. The plots were generated from a deaerated lead mine water and Split Set steel samples. Corrosion current and subsequently general corrosion rate are calculated from the slope of a polarization resistance (linear polarization) plot and the Tafel constants. An example of a polarization resistance plot obtained with deaerated lead mine water and a Split Set steel sample is shown in figure 3.

Results of the series of electrochemical tests to determine effects on the apparent corrosion rate of specimen stabilization times are shown in figure 4. Apparent corrosion rates of specimens in either electrolytically cleaned or abraded only (600-grit SiC paper and ultrasonically cleaned in ethanol) conditions stabilized quickly in deaerated water. Apparent corrosion rates of abraded specimens also stabilized quickly in air-saturated water. However, electrolytic cleaning of specimens tested in air-saturated water resulted in very unstable apparent rates for the first few minutes of immersion. Corrosion rates were relatively stable in all cases after immersion for ~4 min. Corrosion rates reported in table 3 for electropolished specimens in air-saturated waters were obtained with specimens that had been cleaned by abrasion only. All other

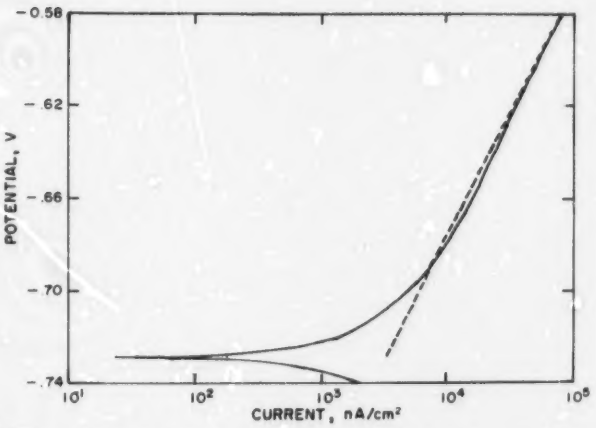

FIGURE 1. - Example of anodic polarization plot. Deaerated lead mine water and HSLA steel.

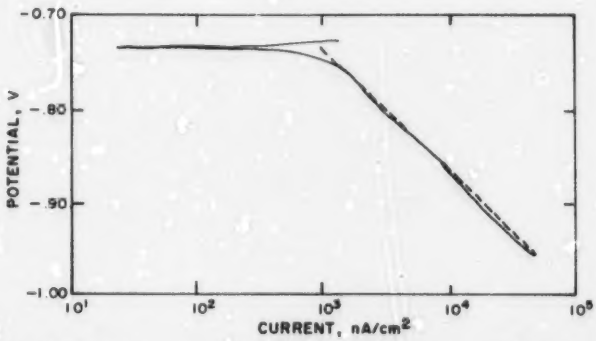

FIGURE 2. - Example of cathodic polarization plot. Deaerated lead mine water and HSLA steel.

TABLE 2. - Analyses of mine waters from Missouri lead and iron mines

Mine	Contents, ppm								pH	Langelier index
	Cations				Anions			Total dissolved solids		
	Ca	K	Mg	NA	Cl	HCO_3	SO_4			
Fletcher.....	13	0.8	22	3.4	24	194	86	265	8.1	-0.2
Indian Creek.....	33	5.4	21	39	105	226	26	451	8.1	+.5
Magmont.....	22	.5	26	1.3	105	269	62	438	8.0	+.2
St. Joe #28.....	18	2.0	19	12	26	213	101	321	8.0	+.3
Pea Ridge[1].....	16	3.8	16	198	29	255	127	565	8.0	+.4

[1]Iron mine; others lead mines.

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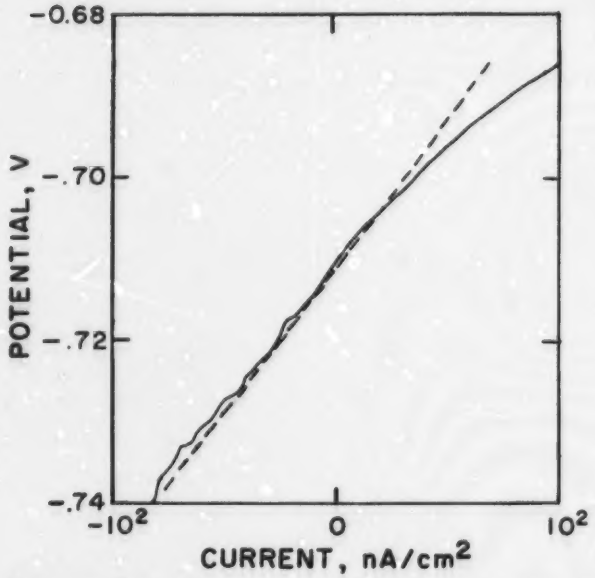

FIGURE 3. - Example of polarization resistance (linear polarization) plot. Deaerated lead mine water and HSLA steel.

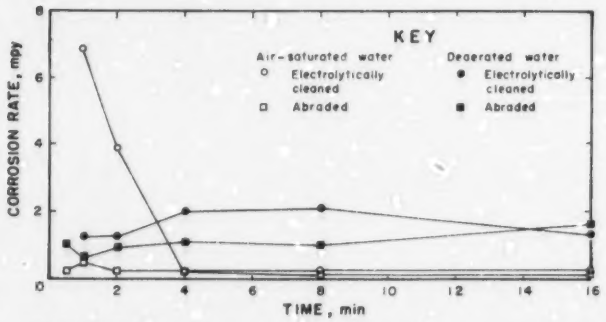

FIGURE 4. - Example of derived electrochemical corrosion rate versus stabilization time for EX-TEN-H60 steel in a lead mine water.

rates were determined on specimens that had been electrolytically cleaned.

Results of electrochemical corrosion tests, shown in table 3 (standard deviation denoted by symbol σ), indicate that KAI-WELL-55 steel exhibits, on the average, slightly lower corrosion rates than EX-TEN-H60. Such a difference was also observed in corrosion studies of these steels in western copper and uranium mine waters (11). Galvanized steel exhibited much lower rates in deaerated waters than either of the two HSLA steels. In aerated waters, however, passivation of both EX-TEN-H60 and KAI-WELL-55 resulted in much lower rates than those of galvanized steel, except in the Pea Ridge water. Passivation was observed only in certain electrochemical tests and was not evident in the gravimetric tests. It may therefore not be a significant factor for the longer term exposure of installed roof bolts. Rates of 17.6 and 11.6 mpy for EX-TEN-H60 and KAI-WELL-55, respectively, in aerated Pea Ridge water were the highest observed. Also, rates for the two steels in deaerated water from this iron

TABLE 3. - Electrochemically determined corrosion rates

Mine water	Test solution temperature, °F	Oxygen content,[1] ppm	Corrosion rate,[2] mpy					
			EX-TEN-H60		KAI-WELL-55		Galvanized	
			Av	σ	Av	σ	Av	σ
Fletcher.....	63	0.4	2.7	0.7	1.4	0.2	0.5	0.1
	63	9.4	.3	.1	.2	.02	2.3	.8
Indian Creek.....	62	.5	1.6	.2	1.5	.2	.3	.05
	62	9.4	.2	.05	.4	.02	3.8	.5
Magmont.....	61	.4	1.8	.3	1.6	.2	.5	.1
	61	9.6	.2	.1	.1	.04	1.6	.2
St. Joe #28[3].....	64	.5	1.6	.4	1.4	.4	.6	.1
	64	9.3	.4	.1	.2	.02	2.3	.9
Pea Ridge[4].....	80	.3	2.8	.3	2.1	.1	.4	.1
	80	7.5	17.6	6.1	11.6	3.3	1.6	.4

[1]Higher values are aerated; those at 0.5 ppm or less are deaerated.
[2]σ is standard deviation.
[3]For same conditions, indicated rates for Swellex steel were 0.1 mpy (deaerated) and 0.2 mpy (aerated).
[4]Iron mine; others lead mines.

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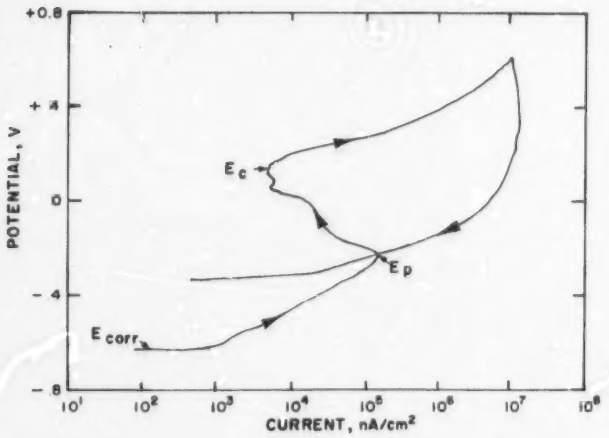

FIGURE 5. - Example of pitting scan showing little tendency of specimen to pit.

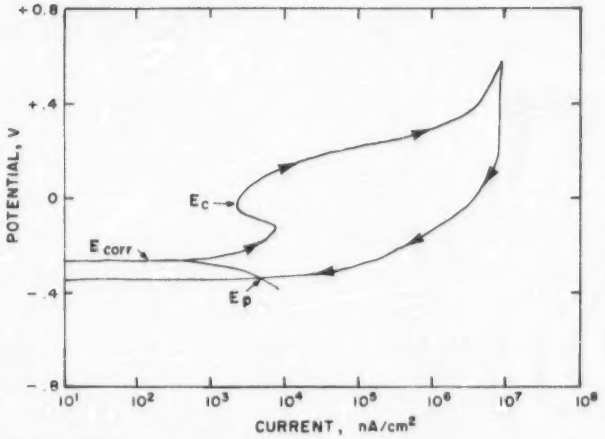

FIGURE 6. - Example of pitting scan indicating tendency of specimen to pit.

mine were higher than rates obtained in the deaerated waters from the lead mines.

Limited electrochemical tests on Swellex bolt steel resulted in indicated corrosion rates lower than those obtained with EX-TEN-H60 or KAI-WELL-55 steels. In deaerated St. Joe #28 water, a rate of 0.1 mpy (σ = 0.02) was observed with Swellex steel compared with rates of 1.6 and 1.4 mpy for EX-TEN-H60 and KAI-WELL-55 steels, respectively, in the same water. Similarly, in aerated St. Joe #28 water, a rate of 0.2 mpy (σ = 0.03) was obtained for Swellex steel compared with rates of 0.4 and 0.2 mpy for EX-TEN-H60 and KAI-WELL-55, respectively.

Corrosion rates determined by the long-term, static weight-loss method are shown in table 4. The slightly lower electrochemical corrosion rates of KAI-WELL-55 steel compared with those of EX-TEN-H60 steel were not evident in the weight-loss tests except in Pea Ridge water, where KAI-WELL-55 steel averaged 1.6 mpy and EX-TEN-H60 averaged 2.3 mpy. The highest rate observed in the weight-loss tests was 2.8 mpy for KAI-WELL-55 steel in Fletcher water. Corrosion rates determined with weight-loss tests in waters containing from 6.3 to 7.2 ppm dissolved oxygen (from air dissolution) were comparable with electrochemically determined rates in deaerated mine waters containing only 0.3 to 0.5 ppm dissolved oxygen. Apparently, rates determined electrochemically in deaerated waters are somewhat more indicative of long-term corrosion rates than those electrochemical rates obtained in air-saturated waters. Passivation was not evident in the static tests, although it may have been a transient occurrence early in the tests on relatively clean surfaces before substantial rust products began to form.

Typical pitting scans of the HSLA and galvanized steels are shown in figures 5 and 6. Figure 5 is a scan indicating little tendency of the specimen to pit.

TABLE 4. - Corrosion rates determined by weight-loss method

Mine water	Test solution temperature, °F	Oxygen content, ppm	Corrosion rate, mpy			
			EX-TEN-H60		KAI-WELL-55	
			Av	σ	Av	σ
Fletcher.....	62	6.3	2.1	0.2	2.8	0.5
Indian Creek....	62	6.4	2.4	.3	2.7	.2
Magmont.....	62	7.2	1.8	.3	1.6	.8
St. Joe #28.....	62	6.7	2.6	.3	2.3	.2
Pea Ridge[1].....	80	6.6	2.3	.4	1.6	.04

[1]Iron mine; others lead mines.

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TABLE 5. - Pitting tendency of HSLA and galvanized steels in Missouri mine waters

Mine	EX-TEN-H60	KAI-WELL-55	Galvanized
PITTING TENDENCY--DEAERATED			
Fletcher	ND	ND	ND.
Indian Creek	Low	High	High.
Magmont	Low	Low	High.
St. Joe #28	Low	Mod	High.
Pea Ridge[1]	High	High	High.
PITTING TENDENCY--AERATED			
Fletcher	Mod	High	ND.
Indian Creek	High	High	High.
Magmont	Mod	High	Low.
St. Joe #28	High	High	Low.
Pea Ridge[1]	High	High	High.

ND Not determined. Mod Moderate.
[1]Iron mine; others lead mines.

If the protection potential (E_p) is more positive than the corrosion potential (E_{corr}), as it is in figure 5, pitting becomes less likely to occur (4, 6) as E_p becomes more positive relative to E_{corr}. E_{corr} is the open-circuit potential. The protection potential E_p is defined as the potential at which the hysteresis loop of the pitting scan is completed and below which (E more negative) pits will not initiate. Figure 6 is a typical plot in which the protection potential is more negative than the corrosion potential and pitting of the specimen is indicated. The pitting potential (E_c, also referred to as the critical potential) has also been used as an indication of pitting tendency, but the protection potential is more reproducible and is now considered a more reliable indicator (6). The pitting potential is defined as the potential at which the current increases rapidly and above which (E more positive) pits will initiate and grow. Research has been done (8) which indicates that, when pit initiation time is considered, $E_c = E_p$.

Results of the evaluation of pitting scans are shown in table 5. Pitting tendency was evaluated on the basis of difference between E_{corr} and E_p. If E_p was more negative than E_{corr}, pitting tendency was rated as high. When E_p was somewhat more positive than E_{corr}, but the difference between E_{corr} and E_p remained in the range 0 to 0.1 V, the pitting tendency was arbitrarily rated as moderate. Similarly, with E_p more positive than E_{corr}, a difference between E_{corr} and E_p greater than 0.1 V was rated as an indication of low tendency to pit.

Moderate to high pitting tendency is indicated for both HSLA steels in all of the air-saturated waters. Passivation of the steels in air-saturated waters during electrochemical testing also indicates a probable tendency to pitting, since this is a common occurrence on metals that passivate. A high tendency to pitting is indicated for galvanized steel in the deaerated waters, as well as aerated Indian Creek and Pea Ridge water. Evaluation of the scans indicates a high pitting tendency for both HSLA steels and galvanized steel in Pea Ridge water in aerated and deaerated conditions.

CONCLUSIONS

Based on results of total-immersion, weight-loss corrosion tests, there is little difference in corrosion rates between EX-TEN-H60 and KAI-WELL-55 steels in Missouri lead and iron mine waters. Slightly lower rates were observed for copper-bearing KAI-WELL-55 steel than for EX-TEN-H60 in electrochemical tests.

Galvanized steel exhibits much lower rates in electrochemical tests in deaerated water (<0.5 ppm dissolved oxygen) than either of the HSLA steels.

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Passivation effects observed in electrochemical tests in air-saturated (>9.3 ppm dissolved oxygen) lead mine waters resulted in very low rates of 0.4 mpy or less for both HSLA steels. Passivation probably does not occur in bolt-rock contact areas of installed stabilizers, owing to lack of oxygen. It may occur on bolt surfaces exposed to ample air and moisture; but, if it occurs on installed stabilizer surfaces, this may be a transient effect for relatively clean surfaces as opposed to rusting surfaces. In the electrochemical tests, passivation was not observed on either of the HSLA steels in the iron mine water. Passivation was also not evident for the two HSLA steels in any of the waters for the total-immersion conditions of atmospheric oxygen dissolution, although it may have occurred to some extent early in these weight-loss tests when the surfaces were relatively free of rust products.

Corrosion rates determined by the long-term weight-loss tests in water at atmospheric oxygen saturation are more comparable with rates determined electrochemically in deaerated water than with electrochemically determined rates in air-saturated water.

Limited electrochemical tests on Swellex stabilizer steel indicated corrosion rates generally of the same order of magnitude as those obtained with the HSLA steels used for Split Set stabilizers.

Moderate to high pitting tendency was observed for both HSLA steels in all air-saturated mine waters. Galvanized steel exhibited a high tendency to pit in all deaerated waters. Pitting tendency was high for both of the HSLA steels and the galvanized steel in either the deaerated or air-saturated iron mine water.

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